

Chemical Analysis of Particulate Matter (PM) in Support of ETD's PM Research Research

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* Introduction. Increasingly, worldwide epidemiological research links PM air pollution with adverse cardiopulmonary health effects and mortality. The specific physical and/or chemical constituents of PM responsible for the observed effects are not yet known.

Growing evidence points to bioavailable transition and heavy metals found in PM. The ability to conduct a complete compositional analysis of PM is a crucial prerequisite for the proper interpretations of results from toxicological studies.

Methods. Our lab's main task is to determine the elemental composition and relative bioavailability of source, urban ambient, and surrogate PM samples. We also measure metal content in all types of biological samples. For new sample types and elements, we research / adapt / adopt / develop methods accordingly. Whenever possible, we use EPA standard methods for sample prep and analysis, to help assure data quality.

ETD sample prep:

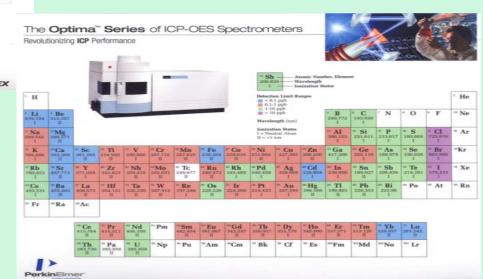
Sample prep usually requires extraction and/or digestion to solubilize the elements of interest into an acidic solution, prior to aspiration into ETD's ICP elemental analyzers. Using Clean Chemistry techniques during preparation of samples and standards extends detection limits of the analyzers – and allows better characterization of smaller (<100ug) PM samples.

ETD methods for elemental analysis:

- Inductively Coupled Plasma Mass Spectrometry (ICP-MS), using EPA Method 6020a
- Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES), using EPA Method 200.7

Together the analyzers cover most of the periodic table, and can measure PM elemental composition from percent to ug/g (parts per million). The detection limit ranges as shown below are generally achievable in practice.

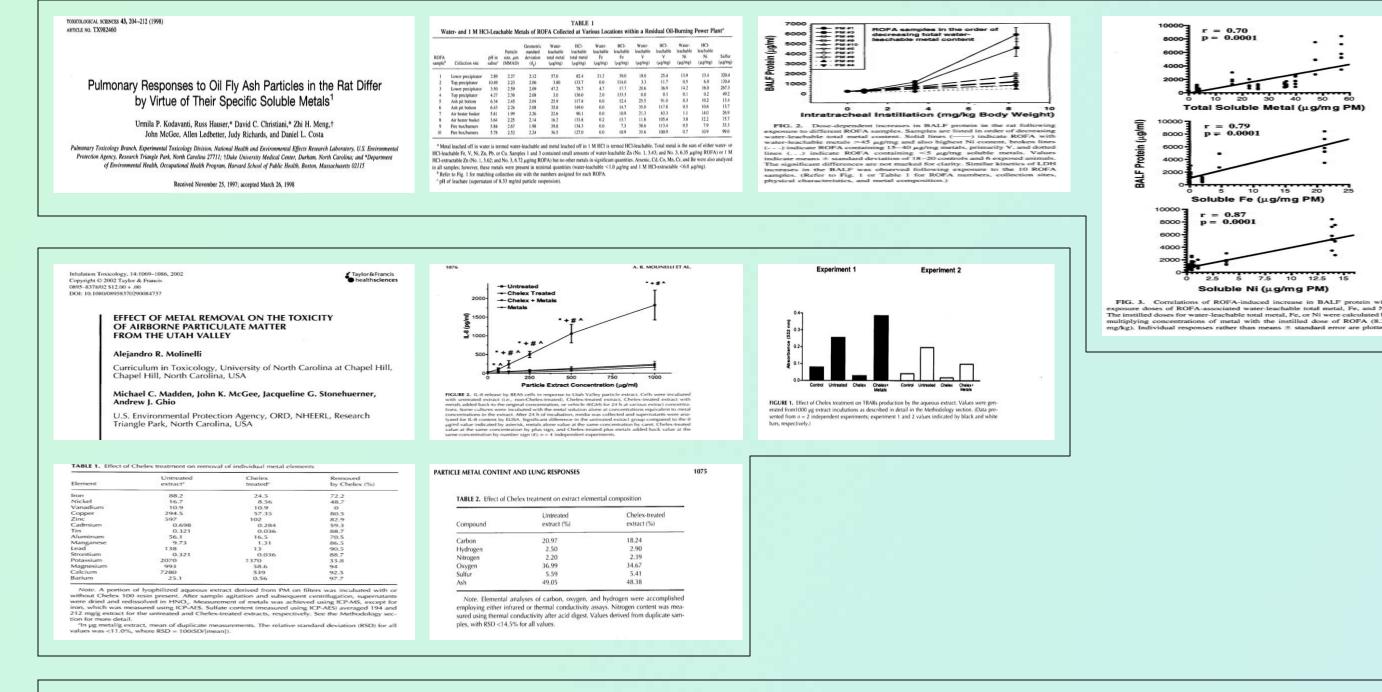




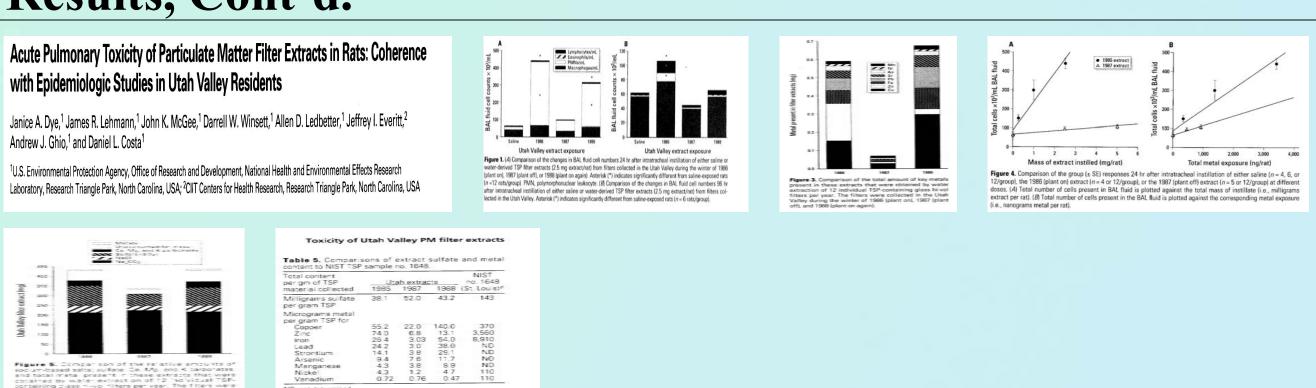
* Methods, Cont'd.

ETD also works with other labs to further characterize PM samples:

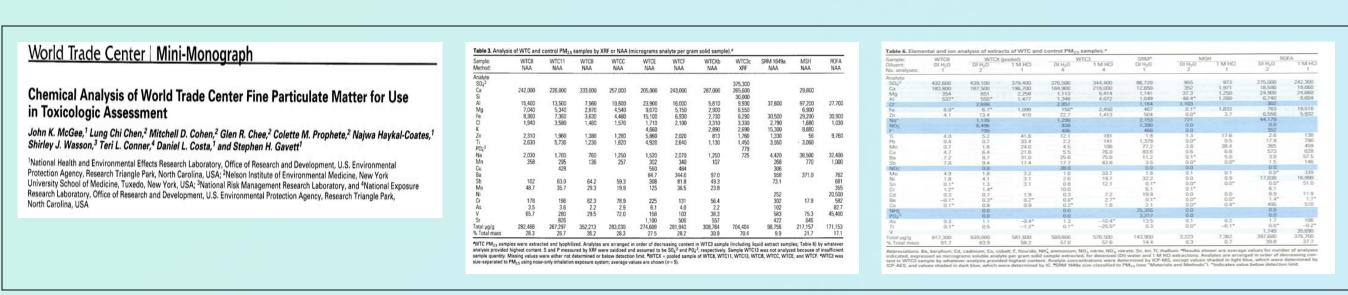
- Carbon speciation analysis organic/elemental/carbonate (Sunset Laboratories)
- Elemental, nitrate, sulfate analysis (Research Triangle Institute, NERL)
- Total elemental content: X-Ray Fluorescence (XRF, NERL, NRMRL), Neutron Activation Analysis (NAA, NC State University)
- Microscopy and elemental composition: Scanning Electron Microscopy Energy Dispersive X-Ray Analysis (SEM-EDX, NERL)
- **Results.** ETD's analytical capability has directly contributed to numerous studies that linked toxic effects with specific PM transition metals. Here are some results:



* Results, Cont'd.



PARTICULATE MATTER



Future Directions.

- Improve and extend our analytical capabilities by adding more elements and upgrading analyzers. For example, during summer 2003 we retired a 15-year-old ICP-OES analyzer (PerkinElmer P40) and replaced it with a state-of-the-art model (PerkinElmer 4300DV). For an equal amount of sample (3.5mL), the new analyzer provides both more elements analyzed (32 vs. 2) and improved detection limits (by a factor of 5 −10 for every element). Reduced sample requirements will allow analysis of more samples using both ICP-MS and ICP-OES analyzers, further strengthening QC and data quality.
- Better characterize PM samples for both total and bioavailable mass fractions. This involves getting more PM mass during sampling, performing both total elemental analysis (XRF, NAA) and bioavailable elemental analysis (ICP, IC), and adding new analytical capability. We are including the following analytes: watersoluble carbon content, ammonium, chloride, and phosphate ion content; total carbon-hydrogen-oxygen (CHO) content.

We will use analytical results from the two bullets above to provide better source apportionment information on PM, and to prepare for upcoming research in Air Toxics.

- Improve speciation of bioavailable elements, by separating water-soluble PM into three fractions: 1) free and loosely-bound, 2) organics-bound, and 3) tightly-bound. The expected degree of metal bioavailability is: free / loosely-bound > organics-bound > tightly-bound.
- Develop stable isotope methods for tracing elements found in the body at high levels, such as iron, copper, and zinc. These samples will all be in biological or tissue culture matrices preconcentration of the metals of interest while removing the sample digest matrix will give more precise isotope measurement.
- Analyze clinical samples for major (Na, Cl, C), minor (Ca, Mg, K, P, Fe, Cu, Zn), and trace (Se) elements to investigate how PM inhalation can affect their interbalance.

SOLVING AGENCY PROBLEMS